



CHEMICAL CALIBRATION: PROVIDERS OF PROFICIENCY TESTING SPECIFIC OPERATIONS CHECKLIST

Instructions to the Assessor: This checklist addresses specific accreditation criteria prescribed in applicable sections of NIST Handbook 150-19.

Place an "X" beside any of the checklist items that represent a deficiency. Place a "C" beside each item on which you are commenting for other reasons. Record the item number and your written deficiency explanations and/or comments on this list or on the comment sheet(s). Place a check beside all other items you observed or verified at the provider's facility.

1 Organization and management

(See General Operations Checklist.)

2 Quality system, audit and review

_____ 2.1 The provider shall have the appropriate versions of the following documents available for reference:

_____ 2.1.1 NIST Handbook 150, *NVLAP Procedures and General Requirements*, March 1994;

_____ 2.1.2 NIST Handbook 150-19, *NVLAP Chemical Calibration: Providers of Proficiency Testing*, June 1999;

_____ 2.1.3 USEPA *National Standards for Water Proficiency Testing Studies: Criteria Document*, December 1998;

_____ 2.1.4 *NELAC Standards*, July 2 1998;

_____ 2.1.5 ISO Guide 30, *Terms and definitions used in connection with reference materials*, 1992;

_____ 2.1.6 ISO Guide 34, *Quality system guidelines for the production of reference materials*, 1996;

_____ 2.1.7 ISO Guide 43, *Proficiency testing by interlaboratory comparisons, Part 1 and Part 2*, 1997;

_____ 2.1.8 ISO/IEC/BIPM, *Guide to the Expression of Uncertainty in Measurement*, 1993; or ANSI/NCSL Z540-2-1997, *U.S. Guide to the Expression of Uncertainty in Measurement*;

_____ 2.1.9 AOAC, *The International Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories*, 1993.

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- _____ 2.2 The provider's quality documentation contains procedures or instructions describing the following:
 - _____ 2.2.1 training of staff and documentation of the performance of analysts and technical staff;
 - _____ 2.2.2 sample custody and handling procedures, and procedures for ensuring the prevention of contamination or degradation of proficiency test materials or their component materials;
 - _____ 2.2.3 equipment maintenance, calibration, and verification;
 - _____ 2.2.4 operation of proficiency tests, including registration of laboratories under test, distribution of materials and instructions, and collection of data;
 - _____ 2.2.5 data processing for proficiency tests, and generation and distribution of reports; and
 - _____ 2.2.6 security of data and reports.
 - _____ 2.3 The provider shall conduct an internal audit at least annually to verify that its operations are in compliance with its quality manual and this program.

3 Personnel

- _____ 3.1 The provider shall ensure that staff members are aware of the extent of their area(s) of responsibility.
- _____ 3.2 The provider shall maintain documentation for each staff member that contains:
 - _____ 3.2.1 staff member's title and description of that job position;
 - _____ 3.2.2 job and quality assurance responsibilities;
 - _____ 3.2.3 résumé;
 - _____ 3.2.4 training;
 - _____ 3.2.5 assigned procedures and duties; and
 - _____ 3.2.6 results of periodic testing performance reviews.
- _____ 3.3 The provider shall have a description of its staff training program including its criteria for successful completion.

- _____ 3.4 Analysts and technical supervisors shall participate in some form of continuing education, such as formal course work, in-house education, and technical meetings, and have access to journals, publications and other information that describe advances in the field.

4 Accommodation (facilities) and environment

- _____ 4.1 The provider shall maintain a facility that:
- _____ 4.1.1 provides a safe work environment for all employees;
 - _____ 4.1.2 permits safe handling of any chemical used for any purpose; and
 - _____ 4.1.3 prevents contamination or degradation of proficiency test materials and of the raw materials from which they are prepared.

5 Equipment and reference materials

- _____ 5.1 The provider shall maintain equipment and reference materials appropriate to the proficiency test materials being prepared and value-assigned.
- _____ 5.1.1 Appropriate Standard Reference Materials from NIST will be available for use, together with the certificates that accompany the SRMs.
 - _____ 5.1.2 SRMs will be properly stored and used according to the instructions given on the certificate.
 - _____ 5.1.3 Analytical and other laboratory equipment will be properly maintained, calibrated, and as necessary, validated together with the analytical methods used by the laboratory.

6 Measurement traceability and calibration

- _____ 6.1 Calibrations, value-assignments, and overall analytical verifications are performed by properly trained staff using Standard Reference Materials traceable to NIST, when available. When NIST certified reference materials are not available, appropriate reference materials certified by other national and international bodies may be used.
- _____ 6.2 Reference materials shall be stored and used according to the instructions given on their certificate and guarded from degradation and contamination during storage and use. Care will be given to verifying that the correct certificate is available for each reference material and that the expiration date given on the certificate for the material has not passed.

7 Calibration and test methods

- _____ 7.1 Starting materials will be verified for identity and assessed for purity or composition as appropriate.



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- _____ 7.2 Corrections for component purity will be applied prior to giving assigned values to proficiency test materials.
 - _____ 7.3 Analyses and test methods may be designed by the provider, but any such methods will have demonstrated overall validations. Methods designed by the provider will produce results of sufficient accuracy to meet the specifications of the proficiency tests for which they are intended.
 - _____ 7.4 Materials to be used as proficiency test materials will be verified with respect to assigned values, uncertainty, homogeneity, stability and suitability for intended use in a given program (e.g., suitability of a PT material/study design for use with program-designated performance evaluation criteria to be used by the provider to evaluate laboratories under test).

NOTE: Traceability to national standards includes traceability to standards maintained or defined at national laboratories in foreign countries, having Mutual Recognition Agreements with NIST, where applicable.

Where applicable, the methodology of the *Guide to the expression of uncertainty in measurement*, 1993, or ANSI/NCCL Z540-2-1997, shall be used as the basis for the expression of uncertainty of the measurement. NIST Technical Note 1297, September 1994, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, is a practical application document written around the *Guide to the expression of uncertainty in measurement*. A guide is also available that deals expressly with analytical chemistry. It is *Quantifying Uncertainty in Analytical Measurements* (English edition), produced by Eurachem and distributed by BSI Customer Services, 389 Chiswick High Road, London, W4 4AL, United Kingdom. Where detailed procedures are not used to quantify and combine uncertainties (i.e., use of analytical accuracy ratio concepts), the sources of uncertainty shall be tabulated and demonstrated to be acceptable for the measurement undertaken.

NOTE: One suitable approach for the homogeneity testing of test items is described in The *International Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories: Appendix II: A Recommended Procedure for Testing Materials for Sufficient Homogeneity*.

- _____ 7.5 Materials will be tested for effects of shipment during proficiency test studies.

8 Handling of calibration and test items

- _____ 8.1 The laboratory shall have a material log system used to uniquely identify proficiency test materials and their components, and to document processing, storage, and use of the materials. {The system will be consistent with applicable USEPA requirements.} The log shall, at a minimum, include:
 - _____ 8.1.1 date of receipt of the material;
 - _____ 8.1.2 the condition of the material;

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- _____ 8.1.3 documentation of acceptance or rejection of material, including reasons in any case of rejection;
- _____ 8.1.4 a unique laboratory identification number for each material and for each test sample, thereof; and
- _____ 8.1.5 the initials of the person making the above entries in the material log book.
- _____ 8.2 Where there is any doubt as to the proficiency test material's suitability for use (e.g., a mismatch between identification and description), the laboratory shall have a procedure for resolving the problem. This action shall be documented.
- _____ 8.3 Upon receipt of raw materials and chemicals to be used in preparing proficiency test materials, any abnormalities or departures from standard condition as prescribed in the relevant procedures shall be recorded. Where there is any doubt as to the material's suitability for use, or where the material does not conform to the description provided, corrective action shall be taken.
- _____ 8.4 The provider shall have documented procedures and appropriate facilities to avoid deterioration or damage to proficiency test materials, during storage, handling, preparation, and analysis; any relevant instructions provided with the material shall be followed. Where materials have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored and recorded where necessary. Where a material or portion of material is to be held secure (for example, for reasons of record, safety or value, or to enable check analyses to be performed later), the laboratory shall have storage and security arrangements that protect the condition and integrity of the secured materials or portions concerned [see also item 4 of this checklist].
- _____ 8.5 The provider shall have documented procedures for the receipt, retention or safe disposal of test materials, including all provisions necessary to protect the organization's integrity.
- _____ 8.6 The provider shall have and use documented procedures for producing proficiency test materials having assigned values for analytes that differ from batch to batch randomly and cover, over time, the required range established by USEPA.

9 Records

- _____ 9.1 The provider's quality system documentation shall have written procedures for the storage and retrieval of records.
- _____ 9.2 Records are stored in a logical fashion allowing retrieval within one working day.
- _____ 9.3 The provider shall have documentation, either electronic backup or "paper" hard copy, to verify survival of original data if computer systems are used for primary data retention.

_____ 9.4 The provider shall ensure that the analyst or proficiency testing professional signs (or initials) and dates the original data.

_____ 9.5 The following records are maintained for a minimum of 5 years:

_____ 9.5.1 materials log;

_____ 9.5.2 original data collected by analyst;

_____ 9.5.3 identity of personnel involved in sample preparation and value-assignment;

_____ 9.5.4 analytical data, including assigned values and uncertainties;

_____ 9.5.5 quality control activities and results;

_____ 9.5.6 proficiency test results of the laboratories under test and summary reports;

_____ 9.5.7 equipment and maintenance;

_____ 9.5.8 test reports; and

_____ 9.5.9 records of all actions taken in response to testing complaints.

10 Certificates and reports

(See General Operations Checklist.)

11 Subcontracting of calibration or testing

(See General Operations Checklist.)

12 Outside support services and supplies

(See General Operations Checklist.)

13 Complaints

(See General Operations Checklist.)

14 Operation of proficiency test studies

_____ 14.1 In addition to the requirements in the General Operations Checklist, the provider will be assessed in the following specifics:

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- _____ 14.1.1 Parameter assigned values with associated uncertainties are established and provided to NIST in a secure manner prior to the distribution of materials for each proficiency test study and are not changed on an ad hoc basis. For cases where values are assigned based on study data, this requirement is not applicable. A description of the PT material composition and any required dilutions by the laboratory under test are also provided to the Analytical Chemistry Division (ACD) of NIST.
 - _____ 14.1.2 All calendar dates set for a given proficiency study are adhered to closely, including completion of data processing and reporting of results.
 - _____ 14.1.3 Reports to all participants are issued on the same day.
 - _____ 14.1.4 Complaints by laboratories under test regarding specific analytical data are resolved in a timely fashion and documented for review by ACD of NIST.
 - _____ 14.1.5 The name, title and signature of the Approved Signatory accepting technical responsibility for the tests and test report, and the secure identification code assigned to the laboratory under test, are available for each laboratory under test.
 - _____ 14.1.6 The name(s) and address(es) of the accrediting body(s) to whom the PT results are to be reported for the specific subject of the proficiency test study are available for each laboratory under test for each study.
 - _____ 14.1.7 All applicable reports are developed and distributed according to current USEPA criteria.
 - _____ 14.2 The provider follows procedures that promote equal challenge among test studies conducted by different providers. Procedures employed will include, but not be limited to, those given below.
 - _____ 14.2.1 Each completed set of test study data will be examined by the provider for anomalous patterns for each analyte. Any analyte that provides anomalous results because of previously unrecognized significant inhomogeneity, instability, inaccurately assigned value, or other loss of integrity of the PT material will not be used to evaluate laboratories under test. At a minimum, the following will be considered:
 - _____ 14.2.1.1 displacement from the expected mean for results of the laboratories under test;
 - _____ 14.2.1.2 unusual dispersion of results;
 - _____ 14.2.1.3 unusual pass-fail results;



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- _____ 14.2.1.4 unexpected changes from earlier tests; and
 - _____ 14.2.1.5 any indication that the challenge of the proficiency test study may have provided a challenge that was either too easy or too difficult.
 - _____ 14.2.2 Any observed anomalies will be described to NIST, in a written study discussion report, together with any indications as to the cause of the anomalies.
 - _____ 14.2.3 The provider cooperates with NIST in any research into, or investigation of, the anomalies that may be necessary.
 - _____ 14.2.4 The provider appropriately identifies in all reports any analyte that provides anomalous study results because of previously unrecognized inhomogeneity, instability, or inaccurate assigned values.
 - _____ 14.3 The provider will have documentation available demonstrating that all applicable shipping and safety regulations are met, and that material distribution is done in a controlled manner, including the provisions that:
 - _____ 14.3.1 Material Safety Data Sheets (MSDS) will be available for all materials that require them and evidence must indicate they are appropriately used as required.
 - _____ 14.3.2 Clear and appropriate instructions to the laboratory under test will be available for all proficiency test studies currently being conducted or that have been conducted during the period of accreditation of the provider of proficiency testing. Instruction sets more than five years old will not be requested by assessors for review.
 - _____ 14.3.3 Material shipment procedures should be checked to assure that adequate consideration is given to protection of material quality and stability.
 - _____ 14.3.4 Shipping records should provide sufficient information to track material custody in the event a recall is required.
 - _____ 14.4 Instructions to laboratories under test will provide adequate guidance to assure that they correctly format and transmit the data that they return. The instructions should include warnings to the laboratory under test that they are not to reveal their results, or any other aspect of the test in which they have participated, to any unauthorized person or other laboratory until the test provider has announced the test study is concluded.
 - _____ 14.5 The data collected in a proficiency test study will be processed according to written procedures and criteria. The procedures will provide:
 - _____ 14.5.1 accurate data processing;

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- _____ 14.5.2 fair and equal treatment of laboratory results; and
 - _____ 14.5.3 clear reporting of laboratory status with respect to the criteria for acceptable performance.
 - _____ 14.6 Results of each proficiency test study will be available in electronic data formats specified by USEPA. For each study the results will be provided in four forms:
 - _____ 14.6.1 study discussion report, available for wide distribution, describing the general outcome of the study and describing any anomalies;
 - _____ 14.6.2 study summary report, available for wide distribution, and revealing no individual laboratory results;
 - _____ 14.6.3 individual laboratory evaluation reports, so coded as to completely obscure which laboratory was the source of the data; and
 - _____ 14.6.4 uncoded laboratory evaluation reports.
 - _____ 14.7 The laboratory must have, and demonstrate adherence to, a plan for distribution and security of proficiency test results. The plan must contain adequate controls that tested laboratories will be assured that their uncoded results will be made available only to authorized recipients.

